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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of Hirotaka Minami et al.

Application No. 10/578,137

Art Unit 1796

Filed: May 3, 2006

Examiner Nathan M. Nutter

For SEALING MATERIAL FOR AUTOMOBILE DOOR

**STATEMENT UNDER 37 CFR 1.125
[SUBSTITUTE SPECIFICATION]**

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

As required in the Office communication mailed March 31, 2009, submitted herewith is a substitute specification, containing no new matter, and a marked-up copy. The presently submitted substitute specification more closely resembles the originally filed specification text than the substitute specification filed 9 January 2009.

Favorable action is requested.

Respectfully submitted,

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SEALING MATERIAL FOR AUTOMOBILE DOOR

TECHNICAL FIELD

[0001] The present invention relates to a sealant for automobile doors, and in particular, to a sealant which is used to bond an inside screen to the inner panel of an automobile door and which is excellent in hot creep resistance and shear adhesive strength at high temperatures, particularly in consideration of the application thereof in summer, since the sealant comprises partially crosslinked synthetic rubber(s) as a rubber component.

BACKGROUND OF THE INVENTION

[0002] A major purpose of sealants of this type is to impart waterproofing to automobile doors, such as by bonding inside screens to the inner panels the doors. Hitherto, the conventional solvent-containing type, butyl rubber-based sealants comprised butyl rubbers, plasticizers, tackifiers, fillers, solvents, etc. and were in the form of a paste at ordinary temperatures. These sealants, however, are lacking in strengths (such as hot creep resistance and shear adhesive strength) at high temperatures (for example 80°C) when considered for use in Summer. Therefore, failure to impart waterproofing to automobile doors—a major purpose of such sealants—was not unusual; the inside screens (generally, films of polyethylene or polyvinyl chloride) tended to peel or shift their positions.

[0003] On the other hand, a solvent-free sealant has been proposed which has a high viscosity at ordinary temperatures. This sealant comprises a polybutene resin, lubricant, tackifier, filler, and a fibrous component and is of hot-applying type, which is discharged suitable for use in an application at a temperature of 40 to 80°C, and the hot-applying-type sealant is used to prevent sealant flow at 80°C or facilitate peeling and recovery of the sealant in the maintenance of automobile doors, because of its low peel strength (cf. JP-A-2000-26836).

DISCLOSURE OF THE INVENTION

[0004] The present inventors have vigorously proceeded with their research in order to provide a sealant for automobile doors which has a dischargeability at a hot applying temperature, is competitive with the above mentioned hot-applying-type sealant, and shows high strength under high temperature. As a result, they have found—and the present invention is accomplished based on such finding—that the desired sealant can be obtained by blending partially crosslinked butadiene-acrylonitrile rubber (NBR) and/or butadiene-styrene rubber (SBR), as a rubber component, with a plasticizer and a filler.

[0005] The present invention provides a sealant for automobile doors, which comprises partially crosslinked NBR and/or SBR, a plasticizer, and a filler and its use in bonding an inside screen to the inner panel of an automobile door.

[0006] The partially crosslinked NBR and SBR to be used in the present invention are prepared by partially crosslinking NBR and SBR with a crosslinking agent such as divinylbenzene or sulfur, respectively, and are commercially available from JSR Co. and Nippon Zeon Co., Ltd.

[0007] The sealant of the present invention may contain an uncrosslinked synthetic rubber, which is not crosslinked at all, in addition to the partially crosslinked synthetic rubber(s). Examples of the uncrosslinked synthetic rubber include acrylonitrile-isoprene rubber (NIR), NBR, SBR, butadiene rubber, isoprene rubber and the like. Especially preferable is a synthetic rubber having a Mooney viscosity of 30 to 50 (JIS K-6300).

[0008] In the present invention, the plasticizer is used to swell and dissolve the above partially crosslinked synthetic rubber(s). Specific examples of the plasticizer include phthalates, fractionated products of petroleum, for example DOP, DBP, DIDP, BBP, DINP and DHP, and higher alcohol phthalates.

[0009] In the present invention, the filler is necessary to adjust the flowability and physical properties of the sealant. Examples of the filler include heavy calcium carbonate, surface treated calcium carbonate, clay, talc, silica powder, cellulose powder, resin powder, metal powder, glass microballoons, plastic microballoons, fibrous filler, acicular filler, etc.

[0010] The sealant for automobile doors according to the present invention comprises the above partially crosslinked NBR and/or SBR, a plasticizer and a filler as essential components. Preferably, the above uncrosslinked synthetic rubber (especially uncrosslinked NBR) is additionally blended to these essential components. Optionally, if needed, commonly used additives such as a pigment, a tackifier, an antioxidant, a crosslinking agent, etc. may be appropriately added.

[0011] In this regard, the total content of the partially crosslinked synthetic rubbers may be selected usually in the range of 5 to 45% (% by weight, unless otherwise specified), preferably 5 to 25%. When this total content is smaller than 5%, the resulting sealant may not have sufficient hot creep resistance. When it exceeds 45%, the dischargeability or workability of the resulting sealant tends to become undesirable.

BEST MODES FOR CARRYING OUT THE INVENTION

[0012] The present invention is explained in more detail by way of an example of the invention and a comparative example.

Example 1

[0013] A partially crosslinked NBR ("DN214" available from JSR Co.) (5.5 parts) (parts by weight, unless otherwise specified) and a partially crosslinked SBR ("Nipol 1009" available from Nippon Zeon Co., Ltd.) (5.5 parts) are formed into sheets, using mixing

rolls, and these synthetic rubbers are mixed and dissolved in DINP (33 parts) with a pressure kneader. Then, heavy calcium carbonate (36 parts) and surface treated calcium carbonate (20 parts) are added, and the mixture is homogeneously dispersed with a kneader to obtain a sealant (100 parts) for automobile doors.

Comparative Example 1

[0014] The butyl rubber base sealant containing a solvent which is in the form of paste at ordinary temperatures is used.

Performance Tests

[0015] The sealants of Example 1 and Comparative Example 1 are subjected to the following performance tests.

(1) SOD Viscosity

[0016] A SOD viscometer is used to measure the SOD viscosity (Pas) of each of the sealants at each of the shear rates (sec^{-1}) and each of the measuring temperatures. This measurement is done when each of the sealants is in the initial condition, after the sealant is stored at 50°C for 5 days, and after the sealant is stored at 80°C for 5 days, respectively. The results are shown in Table 1 below.

Table 1

				Unit: Pa. s				
				Shear rate (sec ⁻¹)				
				10	20	78	200	430
Ex. 1	Initial condition	Measuring temperature	20°C	—	696	294	—	133
			60°C	645	411	155	91	51
	After 50° ×5 days		80°C	560	350	134	73	46
	After 80° ×5 days		60°C	739	459	184	103	61
			60°C	872	510	199	112	63
C. Ex. 1	Initial condition		20°C	525	421	289	226	S.O.
			30°C	335	246	156	124	104
			40°C	283	204	134	103	84

Note) S.O.: scale over

(2) Shear Adhesive Strength

[0017] Each of the sealants is U-like applied to the peripheral edges of a painted steel sheet (hot applying at 60°C in the case of the sealant of Example 1), and a polyester film is laminated on the painted steel sheet. The laminate is cured at 20°C and 65% RH for 2 hours and, then, the shear adhesive strength of the laminate (N/6.25 cm²) is measured at each of the measuring temperatures. The results are shown in Table 2 below.

Table 2

<u>Measuring temp.</u>	<u>Example 1</u>	<u>Comparative Example 1</u>
20°C	4.19 CF	2.15 CF
60°C	3.11 CF	0.58 CF
80°C	2.86 CF	0.50 CF

Note) CF: the cohesive failure of the sealant

(3) Hot Creep Test

[0018] Each of the sealants is so applied to an electrodeposition steel sheet as to form a bead-like layer thereof (10 mm width \times 100 mm length \times 5 mm thickness) (hot application at 60°C in the case of the sealant of Example 1). A polyethylene film of 20 mm width \times 150 mm length is laminated on the bead-like sealant layer, so that the polyethylene film can be projected by just 40 mm in the lengthwise direction from the sealant layer. Then, the laminate is cured at 20°C and 65% RH for 2 hours. After that, a load weighing 0.5 g or 1.0 g is attached to the projected edge of the film, and the test laminate is raised upright so that the load can be on the upper side (while the edge of the film is bent due to the weight of the load). The test laminate in this state is left to stand in an oven at 80°C for 3 hours or 24 hours. After that, the peeling and shift of the film are observed. The results are shown in Table 3 below.

Table 3

<u>Load</u>	<u>80°C</u>	<u>Example 1</u>	<u>Comparative Example 1</u>
0.5 g	3 hrs.	No change	AF: 60 mm <
0.5 g	24 hrs.	No change	AF: 60 mm <
1.0 g	3 hrs.	Cf: 0.5 to 1.0 mm	AF: 60 mm <
1.0 g	24 hrs.	Cf: 0.5 to 1.0 mm	AF: 60 mm <

Notes) AF: an interface failure between the film and the sealant

CF: the cohesive failure of the sealant

mm: the unit of the length of the peeled portion of the film